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APPLICATION OF ROCHROME LIQUID CRYSTAL TAPES FOR THERMOGRAPHIC TESTING OF BONDED STRUCTURES

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Prepared by Hoffmann-La Roche Inc.
Nutley, New Jersey

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ABSTRACT

This report describes the use of ROCHROME liquid crystal tape for thermographic testing of bonded structures for aircraft. The techniques for applying the tape, heating the test panels, and photographing the resulting thermal patterns are described and discussed.

The results obtained are compared with those reported previously for the same test panels coated directly with liquid crystals applied by spraying. Other panels previously examined by various nondestructive techniques, but not by thermography, were also tested. Results for these latter panels are presented and discussed.

For both sets of panels good correlation with previous work was obtained. Deficiencies associated with present ROCHROME tape are described. In general, these stem from the lack of optimization for materials testing of the ROCHROME tape, particularly with respect to the adhesive system and supporting film.

FOREWORD

This report was prepared by Applied Sciences, Technical Development Department, Technical Division, Hoffmann-La Roche Inc., Nutley, New Jersey, under NASA Contract NAS 8-26848, "Thermographic Testing of Bonded Structural Panels". The work was administered under the direction of the Roche Chemical Division by Mr. Martin L. Tanzer.

Technical management was under the direction of Dr. August Sturzenegger and Dr. Arthur Mlodozeniec. Mr. Lawrence White was responsible for technical conduct of the program and he was assisted by Mr. Emil Kopko. Photographic support was under the direction of Mr. Carmen Aurrichio and Mr. Frank Warner.

Lawrence White
Mr. Lawrence White

INTRODUCTION

History:

The first use of liquid crystals for the contact thermographic method of nondestructive testing of bonded structures for aircraft was reported by S. E. Cohen of Lockheed-Georgia Company. This work was performed under NASA Contract NAS 8-20627 entitled "The Application of Liquid Crystals for Thermographic Testing of Bonded Structures". (1) For this project NASA supplied to Lockheed-Georgia 35 test panels fabricated with specific built-in defects representing 52 variations of material and construction types used in aerospace structural composites.

Cohen's report concluded that thermographic testing of bonded structures with liquid crystals is entirely feasible and could offer advantages over other thermographic testing techniques. A conclusion was reached that the use of liquid crystals for testing and recording results is far less expensive than infrared systems. The disadvantages were the limitations imposed by the necessity of formulating and spraying liquid crystals directly on the surface to be tested. In order to provide quality assurance, Lockheed was required to design and use liquid crystal calibrators and other instrumentation for optimum utilization of the technique. For wide-spread applicability of the liquid crystal system, it was recommended by Cohen that the spraying procedure be eliminated and that a means be developed for using removable and reuseable coated films containing the liquid crystals.

Purpose:

In view of the aforementioned results, it was apparent that ROCHROME Liquid Crystal Tape would be a prime candidate for such non-destructive testing. Contract NAS 8-26848 was negotiated in order to compare the performance of ROCHROME with the sprayed liquid crystals used by Lockheed-Georgia. The test panels were to be selected from those used by Lockheed and selected portions of these panels were to be shipped to Hoffmann-La Roche for use in this project. In addition, evaluation was to be made of ROCHROME for other types of composites applicable to Space Shuttle requirements, such as the boron epoxy and the graphite epoxy filamentary composites. These latter panels were to be selected from those reported by Pless, Weil, and Lewis (2) of Lockheed-Georgia under Contract NAS 8-25679.

ROCHROME Tape:

ROCHROME Tape is a pressure-sensitive adhesive tape which contains liquid crystals in a protective matrix backed and supported by black plastic film. The composition of the liquid crystal material is variable in order to provide various ranges and sensitivities of thermographic response. The function of the black film is to allow the play of colors to be easily observed by reflected light.

TEST PROCEDURES

Panel Preparation:

The panels were cleaned by wiping with acetone, sprayed with silicone release compound, and then coated on the near and far sides with continuous sheets of ROCHROME Tape. Prior to application, some solvent evaporation from the tape was achieved by air drying at room temperature for approximately one minute after removal of the tape from its substrate of silicone-treated release paper. Regardless of the care taken in application of the tape to the panel, bubbles were found between the tape and the panel. Because of the poor heat transfer between tape and panel at a bubble, a temperature differential was created during the heating and cooling of the panel. This produced a false indication of panel temperature at the bubble. Also, additional bubbles were found to have been formed after the panel had been heated and cooled. The number of bubbles could be reduced, however, by allowing solvent from the tape adhesive to evaporate. In addition, this procedure resulted in better release of the tape from the panel. Gas bubbles trapped between the tape and the panel were removed by pricking the tape with a needle to allow the gas to escape and pressing the tape to the panel to make it adhere at the bubble site. Such bubble removal was done immediately before photography was started in order to eliminate bubbles which developed in the time interval between tape application and photography.

Preliminary Testing:

Before making photographic records, visual observations were made of the thermographic images produced during heating and cooling. For this the radiation source consisted of three Sun Guns (quartz-iodide, tungsten filament lamps, 640 watts each, in reflector mounts) mounted side-by-side in a straight line and aimed so as to irradiate uniformly the panel being tested. A piece of Transite (cemented asbestos) board covered with ROCHROME Tape was used as a test panel to establish uniformity of irradiation by means of the temperature response of the tape. This preliminary observation was done to gain familiarity with the appearance of the thermal images of defects and thus to program properly the sequence of taking photographs of the thermographic images.

Heating and Photography:

The panels were photographed by means of a copying camera set up in the Photo Laboratory. The same lamps were used for irradiation as for photography (four quartz-iodide lamps, 650 watts each, color temperature 3200°K, in reflector mounts). The lamps were symmetrically arranged to irradiate the panels uniformly with the light beams incident on the panel at approximately 45°. The axis of the camera was perpendicular to the face of the test panel. Kodacolor (negative) film was used; color prints made from these negatives were color-balanced with the aid of a color test strip which had been photographed under the same conditions (lighting, distance, exposure, etc.) as the NASA test panels.

The procedure used in heating and photographing the panels was as follows: The panel under test was mounted on a stand with the face of the panel in a plane perpendicular to the floor and to the horizontal axis of the camera and irradiating system. The camera was focused and camera controls (aperture and exposure time) were set. Then, the quartz-iodide lamps were turned on and a stop watch was started. The panel was observed as the thermographic images developed. At suitable times photographs were taken to record these images as the face of the panel heated up through the color transition range of the liquid crystal tape. In most cases photographs were made only during the heating of the panel rather than during cooling. The irradiation time corresponding to each photograph was recorded. The panels were allowed to equilibrate thermally with the ambient room air before carrying out the photography. (The room was air-conditioned but it was necessary to turn off the air-conditioner blower during heating and photography to avoid drafts which would have perturbed the thermographic images.) Other perturbations were associated with edge effects caused by heat flow to clamps and supports used to hold the panels in position. After one face of a panel had been photographed in this manner, the panel was allowed to equilibrate thermally before photographing the other face of the panel. In practice this usually meant that all the near sides of the panels being tested were photographed and then in the same order of panels, all the far sides were done.

TEST RESULTS

I. First Lot of Six Panels from Cohen's Work

A. Results with ROCHROME 2935:

The first lot of six panels (designated IA, IB, IC, ID, IE, and IF) was received from NASA together with photographs from the NASA report by S. E. Cohen. These panels, approximately 8 inches by 24 inches in size, had been cut from the 24 inch by 24 inch panels used in the work described by Cohen. The face sheets of these first panels were aluminum alloy and between the face sheets were phenolic honeycomb cores. According to Cohen's report, this type of panel construction is the most difficult in which to detect defects.

ROCHROME 2935 Tape was applied to both faces (near side and far side) of the panels. The panels were observed during several heating and cooling cycles but no defects could be detected. It is significant that these panels contain only the smallest defects of those present in the original 24 inch by 24 inch panels. Moreover, panels from this series were declared by Cohen to be the most difficult in which to detect defects. It was evident that these first panels were the least desirable to use in developing the test procedure; therefore, it was decided to return these panels to NASA with the proviso that they could be re-examined later. Request was made of NASA to supply us with panels having more easily detectable defects.

II. Second Lot of Eight Panels from Cohen's Work

A. Results with ROCHROME 2935:

The second lot of eight panels from those used by Cohen in his work included IIIA-1, IIIC-1, VB-1, VB-2, VC-2, VD-1, VD-2, IB-Ti, and IIA-Ti. Again, it should be noted that except for the titanium panels (IB-Ti and IIA-Ti), the panels received were selected portions which had been cut from the 24-inch by 24-inch panels used by Cohen. These panels were cleaned and coated with ROCHROME 2935 Tape in the manner already described. Heating and photographing the panels were done as has been described. The photographs obtained were compared with those from Cohen's work. Without making a detailed comparison, it was immediately evident that the ROCHROME 2935 Tape was not performing as well as the sprayed liquid crystals used by Cohen. However, the liquid crystals used by Cohen had a temperature range (red to blue transition) of approximately 1°C, while ROCHROME 2935 Tape has a range of about 5°C. This marked difference in temperature response was evidentally the cause of the poor performance of that tape. Therefore, it was decided to use ROCHROME 3538 Tape, which has a narrower range than ROCHROME 2935 Tape.

B. Results with ROCHROME 3538:

The ROCHROME 2935 Tape was removed from the panels. Removal of the tape from the titanium panels was extremely difficult but no trouble was encountered with the remaining panels. Laboratory trials (without photography) indicated that the ROCHROME 3538 Tape was unquestionably superior to the ROCHROME 2935 Tape. The photographs of these eight panels during heating are shown in Figures 1 to 19.

III. Seven Panels from Work by W. M. Pless, B. L. Weil, and W. H. Lewis:

The panels in this group are designated 101, 105, 208, 210, 422, 525, and 529. These panels were constructed from boron or graphite fibers in epoxy resin matrices and laminated with epoxy resin. The thicknesses of the laminates were stepped at 2 inch intervals. These laminates were then either bonded to titanium strips or to honeycomb cores. During this fabrication process, various defects and fabrication variables were intentionally introduced into these test panels. In the work done by Pless, Weil, and Lewis (2), there had been no thermal thermographic imaging.

For the work reported here, the preparation of the panels and preliminary examination were carried out as already described. In general, adhesion of the tape was poor for these panels; this was particularly so at the boundaries of the steps. These panels were coated with ROCHROME Tape only on the sides bearing their identifying numbers, that is, on the near side of each panel.

A. Results with ROCHROME 3538:

Photographs of thermographic images for these panels during heating are shown in Figures 20 to 28. Discussion of these images is given in the captions for the photographs.

IV. Exploratory Testing with ROCHROME 4748:

After results had been obtained with ROCHROME 3538 Tape, selection was made of areas of some panels to be covered with ROCHROME 4748 Tape. (This tape has a transition temperature of 48.0°C to 49.4°C.)

The panels chosen (IB-Ti, IIA-Ti, 208, 527) were covered with small strips of ROCHROME 4748 Tape over the previously applied ROCHROME 3538 Tape and additional heating and photography were carried out. It was found that the normal arrangement of lights was inadequate to heat the ROCHROME 4849 Tape to its transition temperature; therefore, the lights were moved closer to the test panels and photographs were made.

This high temperature tape was noted to be much more sensitive to ambient conditions. Air drafts and temperature gradients to panel clamps caused recognizable artifacts in the thermographic images. Surrounding the test panels with a heated enclosure might be necessary to eliminate such artifacts in future work.

The photographs are shown in Figures 29 to 32. For three of the panels tested (IIA-Ti, 208, 527), additional thermographic image detail was provided by the high temperature tape. For the remaining panel (IB-Ti) there appeared to be no distinct improvement.

V. Comparison of Results with ROCHROME 3538 with Those of Previous Work:

A. Panels Used in Cohen's Work (1):

The photographs of the thermographic images shown in Figures 1 to 19 were compared with the corresponding photographs accompanying Cohen's report. The results are tabulated in Table I, which indicates for each panel the location, size, and shape of the built-in defect found by Cohen and the presence or absence of the thermographic image in the photograph obtained here.

Table I shows that very good correlation with Cohen's results was obtained for Panels IIIA-1, IIIC-1, IB-Ti, and IIA-Ti; fair correlation for Panels VB-2, VC-2, VD-2; poor correlation for Panel VC-1; and very poor correlation for Panel VB-1. It should be noted that in the panels for which the correlation was rated poor or very poor (VC-1, VB-1), all the defects were 0.75 inch and smaller. For one panel (near side of VD-2) defects were found at Roche which had not been detected in Cohen's work.

B. Panels Used in Work of Pless, Weil, and Lewis (2):

Evaluation of the results obtained (Figures 20 to 28) for the panels constructed of graphite or boron fibers in epoxy matrices could not be made by direct comparison of thermographic images, since no thermal imaging results had been obtained by Pless, Weil, and Lewis. Instead, the results of the thermographic imaging obtained here are given in Table II for comparison with the results of nondestructive testing (ultrasonic C-Scan and x-ray radiography) by Pless, Weil, and Lewis. Detailed comparison has not been made because the liquid crystal tape thermographic images are visually so different from the ultrasonic and x-ray recordings. As the table indicates, the liquid crystal tape produced thermographic images which reveal many of the defects reported by Pless, Weil, and Lewis; however, quantitative comparison is not possible.

CONCLUSIONS

ROCHROME Liquid Crystal Tape has been shown to be applicable to the thermographic testing of bonded structures. The results are similar in quality to those obtained by means of liquid crystals sprayed directly on the surfaces to be tested. In some instances small defects detected by sprayed liquid crystals were not detected by ROCHROME Liquid Crystal Tape. This may be attributable to differences in thermographic response and thermal conductivity between the two systems (sprayed liquid crystals and ROCHROME Liquid Crystal Tape).

Comparison with other nondestructive test methods (ultrasonic C-scan and x-ray radiography) further demonstrated the applicability of ROCHROME Liquid Crystal Tape.

RECOMMENDATIONS

It is recommended that ROCHROME Liquid Crystal Tape systems be optimized for use in materials testing. The following modifications are indicated:

1. The adhesive system should be made to be compatible with structural materials such as metals, ceramics, plastics, and composites. Good adhesive and facile release are essential, as well as absence of trapped gas bubbles.
2. The black plastic film should have good heat transfer properties. It should have sufficient elasticity and resistance to plastic yield to prevent permanent deformation under conditions of application, testing, and removal.

3. The thermal response of the liquid crystals should be optimized to provide higher thermographic sensitivity.
4. The coated film should be highly uniform and free from artifacts which could be mistaken for defects in the structure under test.

BIBLIOGRAPHY

(1) Cohen, S. E., "The Application of Liquid Crystals for Thermographic Testing of Bonded Structures," NASA CR-88196, PD 2-7, prepared under Contract No. NAS 8-20627 by the Proficiency Development Laboratory, Quality Engineering Department, Lockheed-Georgia Co., Marietta, Georgia, for Marshall Space Flight Center, NASA.

(2) Pless, W. M., Weil, B. L., and Lewis, W. H., "Development, Fabrication, Testing, and Delivery of Advanced Filamentary Composite Non-Destructive Test Standards," NASA CR-61340, prepared under Contract No. NAS 8-25679 by the Materials Development Laboratory, Lockheed-Georgia Co., Marietta, Georgia, for Marshall Space Flight Center, NASA.

Table I

Defects Observed by Cohen in Portions of Panels Sent to Roche

Panel	Side Observed	Location	Size, Inches	*Shape	**Observed at Roche	Figure Number
IIIA-1 (Fiberglass Face Sheets, Near Side 0.012 in., Far Side 0.025 in., Fiberglass Honey-comb, 1 in. thick, 3/16 in. Cell)	Near		Core Splice		+	
		2A	0.25	T	+	
		2B	0.75	C	+	1
		3B	0.25	C	+	
		3C	0.75	C	+	
IIIA-1	Far	2A	0.25	T	+	
		2C	0.50	S	+	
		3A	0.50	C	+	2
		3C	0.75	C	+	
			Core Splice		+	
IIIC-1 (Panel Construction Same as IIIA-1 Except Face Sheets; Near Side 0.037 in., Far Side 0.12 in.)	Near	2A	0.25	T	-	
		2B	0.75	C	+	
		3B	0.25	C	+	3
		3C	0.75	C	+	
			Core Splice		+	
IIIC-1	Far	1B	0.50	S	-	
		1C	0.75	T	-	
		2A	0.25	T	+	
		2C	0.50	S	+	4
		3C	0.75	C	+	
		3A	0.50	C	+	
		4C	0.50	S	+	

*Shape: S = □
T = △
C = O

**Observed at Roche: (+ = Yes)
(- = No)

Table I (cont'd.)

Defects Observed by Cohen in Portions of Panels Sent to Roche

Panel	Side Observed	Location	Size, Inches	*Shape	**Observed at Roche	Figure Number
VB-1 (Fiberglass-Fiberglass Laminate; No Core, Near Side 0.025 in., Far Side 0.037 in.)	Near	1C[a]	0.75	T	-	
		3B	0.25	C	-	
		3C	0.75	C	-	5
		4A	0.75	S	-	
		4B	0.25	C	-	
		4C	0.50	S	-	
VB-1	Far	4C	0.50	S	-	
		1B	0.50	S	-	6
		1C	0.75	T	-	
VB-2 (Cut from Same 24 in. X 24 in. Panel as VB-1)	Near	1D[b]	1.00	C	-	
		1E[b]	1.25	C	-	
		1F[b]	1.50	T	-	
		2D	1.00	S	-	
		2E[b]	1.25	C	-	
		2F	2.00	C	+	7
		3D	1.00	T	-	
		3E	1.25	S	-	
		3F	1.75	S	-	
		4D	1.25	C	+	
		4E	1.00	C	+	
		4F	1.75	S	+	
VB-2	Far	4D	1.25	C	+	
		4E	1.00	C	-	
		4F	1.75	S	+	
		1D	1.00	C	-	
		1E	1.25	C	-	8
		1F	1.50	T	-	
		2E	1.25	C	+	
		2F	2.00	C	+	
		3D	1.00	T	+	
		3E	1.25	S	+	
		3F	1.75	S	-	

*Shape: S = □
T = △
C = O

**Observed at Roche: (+ = Yes)
(- = No)

(a) This defect does not appear in photographs in Cohen's report.

(b) These defects are barely visable in photographs in Cohen's report.

Table I (cont'd.)

Defects Observed by Cohen in Portions of Panels Sent to Roche

Panel	Side Observed	Location	Size, Inches	*Shape	**Observed at Roche	Figure Number
VC-1 (Fiberglass-Fiberglass Laminate, No Core, Near Side 0.037 in., Far Side 0.012 in.)	Near	3C	0.75	C	−	
		4A	0.75	S	+	9
		4C	0.50	S	−	
VC-1	Far	4A	0.75	S	+	
		4C	0.50	S	+	
		1C	0.75	T	−	
		3A	0.50	C	−	10
		3B	0.25	C	−	
		3C	0.75	C	+	
		2B	0.75	C	−	
		1B	0.50	S	−	
VC-2 (Cut from Same 24 in. X 24 in. Panel as VC-1)	Near	1D	1.00	C	−	
		1E	1.25	C	−	
		1F	1.50	T	−	
		3D	1.00	T	−	
		3E	1.25	S	+	11
		3F	1.75	S	−	
		4D	1.25	C	+	
		4E	1.00	C	+	
		4F	1.75	S	−	
		2F	2.00	C	+	
VC-2	Far	4D	1.25	C	+	
		4E	1.00	C	+	
		4F	1.75	S	−	
		2D	1.00	S	−	
		2E	1.25	C	−	
		2F	2.00	C	+	12
		1D	1.00	C	−	
		1E	1.25	C	−	
		1F	1.50	T	−	
		3D	1.00	T	−	
		3E	1.25	S	+	
		3F	1.75	S	+	

*Shape: S = □
T = △
C = O

**Observed at Roche: (+ = Yes)
(− = No)

Table I (cont'd.)

Defects Observed by Cohen in Portions of Panels Sent to Roche

Panel	Side Observed	Location	Size, Inches	*Shape	**Observed at Roche	Figure Number
VD-1 (Fiberglass-Fiberglass Laminate, No Core, Near Side 0.050 in., Far Side 0.025 in.)	Near	-	None	-	None	13
VD-1	Far	-	None	-	None	14
VD-2 (Cut from Same 24 in. X 24 in. Panel as VD-1)	Near	3D	1.00	T	-	
		4E	1.00	C	+	
		2F (c)	2.00	C	+	15
		3F (c)	1.75	S	+	
		4D (c)	1.25	C	+	
		4F (c)	1.75	S	+	
VD-2	Far	2F	2.00	C	+	
		3D	1.00	T	-	
		3E	1.25	S	-	
		3F	1.75	S	-	
		4D	1.25	C	+	16
		4E	1.00	C	+	
		4F	1.75	S	+	
		1D	1.00	C	-	
		1E	1.25	C	-	
		1F	1.50	T	-	
IB-Ti (Titanium Face Sheets, 5 Al-2.5 Sn, Near Side 0.025 in., Far Side 0.032 in., Heat Resistant Phenolic Honeycomb Core 1 in. Thick, 3/16 in. Cell)	Near	1C	0.75	T	+	
		1D	1.00	C	+	
		1E	1.25	C	+	
		1G	1.75	S	+	17
		1H	2.00	C	+	
		2E	1.25	C	-	
		2F	2.00	C	-	
			Core Splice		+	

*Shape: S = □
T = △
C = O

**Observed at Roche: (+ = Yes)
(- = No)

(c) These defects were not detected by Cohen but were detected by Roche.

Table I (cont'd.)

Defects Observed by Cohen in Portions of Panels Sent to Roche

Panel	Side Observed	Location	Size, Inches	*Shape	**Observed at Roche	Figure Number
IB-Ti	Far	1D	1.50	T	+	
		1E	1.25	C	+	
		1F	1.50	T	–	18
		1H	2.00	C	+	
			Core Splice		+	
IIA-Ti (Titanium	Far	1A	0.25	C	–	
Face Sheets,		1B	0.50	S	+	
5 Al-2.5 Sn,		1C	0.75	T	+	
Near Side 0.025 in.		1D	1.00	C	+	
Far Side 0.032 in.,		1E	1.25	C	+	
No Core)		1F	1.50	T	+	
		1G	1.75	S	+	
		1H	2.00	C	+	19
		2B	0.75	C	–	
		2C	0.50	S	–	
		2D	1.00	S	+	
		2E	1.25	C	+	
		2F	2.00	C	+	
		2G	1.75	T	+	
		2H	1.50	S	+	

*Shape: S = □
T = △
C = O

**Observed at Roche: (+ = Yes)
(– = No)

Table II

Boron/Epoxy and Graphite/Epoxy Panels

Panel	Defects Observed by Pless, et al	Defects Observed by Roche	Figure Number	Comments
101	Porosity Resin Variations	Porosity Resin Variations Laminate Structure	20	Tape Adhesion Incomplete
105	Porosity	Porosity; Large Unknown Defect at B_L, 0.060	21	-
208	Inclusion	Porosity; Vertical and Horizontal Streaks; Defect at lower-left hand corner of label	22	Tape Artifacts
210	Inclusion	Inclusion; Some Porosity	23	Poor Tape Adhesion
422	Broken Fibers; Overlap; Spacing Void	Overlap; Some Voids	24	-
[illegible]5	Delamination; Disbond-Titanium; Pre-aged Area	Few Disbonds-Titanium	25	-
527	Delamination; Crushed Core; Disbond-Honeycomb; Pre-aged Area	Crushed Core; Some Disbonds	26	-
528	Delamination; Crushed Core; Disbond-Honeycomb	Some Disbonds	27	-